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Research and Development

# **SEPA** Project Summary

# Chlorinated Organic Compounds in Digested, Heat-Conditioned, and Purifax-Treated Sludges

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A study was conducted to investigate the effects of sludge stabilization methods on the production of priority pollutants and chlorinated organics. Three stabilization methods were examined in pilot studies— the Purifax\* process, anaerobic digestion, and heat conditioning.

Results showed that sludge processed by the Purifax process at chlorine dosages normally used in processing wastewater sludges contains 2 to >14 times the total organic chlorine in raw sludge. Both insoluble and soluble forms of organic chlorine increase with chlorine dosage. According to the study, most of the chlorinated compounds are associated with the solid fraction of the sludge. More than 94 percent of the total organic chlorine is insoluble, and about 97 percent of that is associated with the cake. Using the Tchebychev inequality, it was determined that the probability that Purifax treatment produces the same concentration of insoluble organic chlorine in sludge cake as the other stabilization processes is less than 22.5 percent (assuming a unimodal distribution, this probability is less than 10 percent). Leachate obtained by the EPA extraction procedure contained less than 0.2 percent of the organic chlorine in the sludge cake after Purifax treatment. In analyses for priority pollutants, less than 16 percent of the chlorinated material was identified. A new analytical technique using ionization/gas chromatography/mass spectrometry gave results inconsistent with the priority pollutant analyses.

This Project Summary was developed by EPA's Municipal Environmental Research Laboratory, Cincinnati, OH, to announce key findings of the research project that is fully documented in a separate report of the same title (see Project Report ordering information at back).

#### Introduction

In the Purifax process, chlorine gas is added to wastewater sludge, septage, or digester supernatant to stabilize and condition the material before dewatering and disposal. The chlorine dosage is high (50,000 mg/kg of dry solids), and the residual chlorine concentration is also high (roughly 60 to 400 mg/L). Because of these high concentrations, concern has existed that the process might produce unacceptable levels of chlorinated organics. The objective of this study was therefore to determine the effects of stabilization processes on the production of priority pollutants and chlorinated organics. Three stabilization methods were examined--the Purifax process, anaerobic digestion, and heat conditioning.

# Procedures Sources of Sludge

To allow for variability in sludges from plants receiving different types of wastewater, sludges were tested from three wastewater treatment plants-Amherst, Massachusetts, Willimantic, Connecticut; and Pittsfield, Massachusetts The Amherst plant serves a

<sup>\*</sup>Mention of trade names or commercial products does not constitute endorsement or recommendation for use

residential area that has developed around several college campuses. The Willimantic plant receives flow from some 20 industries, which constitutes about 25 percent of the total flow for the plant. Industrial processes include dyeing of cotton and synthetics, production of woolen fabrics, and poultry processing. The Pittsfield plant receives about 5 percent of its total flow from two industries--General Electric and the Crane Paper Company. The General Electric plant discharges pretreated wastes containing phenolic compounds. The Crane Paper Company's main discharge consists of liquids from a pulp digestion process.

## Anaerobic Digestion

The feed material added to the batch digesters included mixtures of raw sludge and digested sludge seed in two ratios of raw sludge to seed by volume, 25/75, and 50/50.

Digestion temperature was maintained at 35°C, and the digester contents were continually mixed with digester gas. The tests lasted 28 days. Gas production was very low by that time, and changes in total volatile solids were minimal. Also, the total volatile solids in the sludge approached the concentration in the digester seed.

#### Heat Treatment

Bench-scale heat-conditioning tests were conducted on a 1-L autoclave. This unit was used as a batch heat-conditioning system in which approximately 0.8 L of sludge was mixed and heated for 30 min to 180°C at 970 kPa. (Full-scale heat-conditioning units operate at 120° to 220°C and 1700 to 2000 kPa for 20 to 30 min.)

## **Purifax Pilot Tests**

The pilot plant tests were conducted to provide sludges with high, medium, and low chlorine residuals (as compared with medium residuals at full-scale Purifax processing plants). Medium residuals were those from 150 to 200 mg/L; high residuals were 280 to 350 mg/L; and low residuals were 60 to 120 mg/L.

### Results

Only Purifax-treated sludges showed substantial increases in total organic chlorine after processing (Figure 1). In these tests and others, the total organic chlorine content in Purifax-treated sludge is 2 to >14 times that in raw, heatconditioned, or digested sludge. The

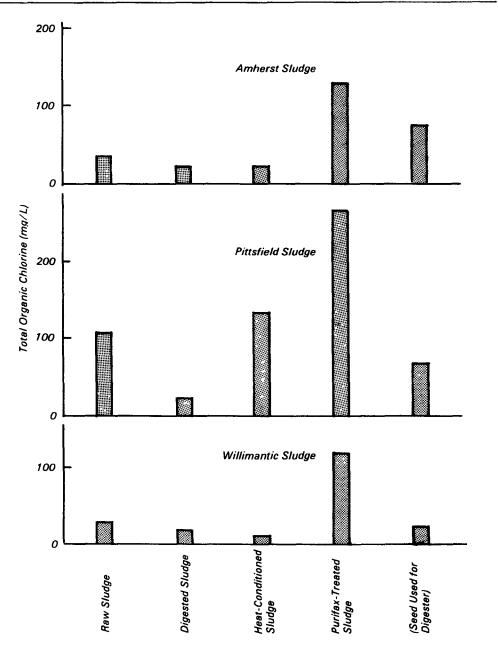


Figure 1. Effects of stabilization processes on total organic chlorine levels in sludge.

only exception is the concentration shown for heat-conditioned sludge from Pittsfield, which appears to be an error. These ratios are for a chlorine residual of about 170 mg/L (a medium residual), which occurred after a chlorine dosage of about 1500 mg/L. The dosage and residual are comparable to those used at Purifax plants.

The concentrations of soluble and insoluble organic chlorine increase with increased chlorine residual or chlorine dosage. There is scatter to the data for all

four combinations, but a statistically significant line of fit can be drawn through the data. Coefficients of determination (r²) range from 0.18 to 0.32. For the number of samples in each case, the trends are significant at 1.5- to 8.1-percent levels.

The tests showed that more than 94 percent of the total organic chlorine in all tested sludges is insoluble. For Purifax-treated sludge, more than 97 percent of the organic chlorine is insoluble. Since most of the organic chlorine is insoluble,

nearly all of it ends up in the cake after centrifuging. The data show that less than 3 percent of the total organic chlorine in processed sludge is in the centrate. In the Purifax-treated samples, only 1.4 percent of the chlorine was in the centrate. And of the portion in the cake, less than 0.2 percent was leached by the EPA extraction procedure.

Tests were conducted to quantify and identify chlorinated compounds present before and after processing in sludge from the Amherst wastewater plant (the one serving a residential area). Test methods included those for quantifying organic priority pollutants and a new analytical technique--ionization/gas chromatography/mass spectrometry. The tests for priority pollutants identified only a portion of the chlorinated compounds. For sludge samples, tests identified less than 16 percent of the chlorinated material in feed sludge and less than 2.6 percent in Purifax-treated sludge. For cake samples, tests identified less than 0.1 percent of the residual chlorinated compounds. Though the new analytical technique identified compounds with properties similar to those of some chlorinated priority pollutants, the two techniques identified different compounds.

Purifax treatment of the Amherst sludge produced an approximate 5-fold increase in total chlorinated organics. It appears that the identifiable chlorinated organic compounds are approximately the same before and after Purifax processing, although proportions have shifted. The reliability of the analytical technique for quantifying the various organic compounds is not sufficiently trustworthy to permit statements about quantitative changes in these compounds. Confirming work is recommended, preferably with improved extractions and analytical techniques.

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James A. Ryan is the EPA Project Officer (see below).

The complete report, entitled "Chlorinated Organic Compounds in Digested, Heat-Conditioned, Purifax-Treated Sludges," (Order No. PB 84-212 695; Cost: \$13.00, subject to change) will be available only from:

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